1977 Subtropical Food Technology Conference

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LANDMARK MOTOR LODGE

WINTER HAVEN, FLORIDA

U. S. CITRUS AND SUBTROPICAL PRODUCTS LABORATORY

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SOUTHERN REGION — FLORIDA ANTILLES AREA

AGRICULTURAL RESEARCH SERVICE

U. S. DEPARTMENT OF AGRICULTURE

PREFACE

The Annual Subtropical Food Technology Conference is sponsored by the Southern Region, Florida Antilles Area, of USDA's Agricultural Research Service. It reports developments in the broad areas of processing, marketing, nutrition, pollution abatement and related subjects, and provides for exchange of information that will benefit the industry, future research and American consumers.

This report summarizes the statements of the various speakers during the Conference. Many of these are progress reports and are subject to change as studies are completed. Please contact authors for latest results before using these reports as a reference.

Dean F. Davis, Area Director

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PROGRAM

1977 SUBTROPICAL FOOD TECHNOLOGY CONFERENCE

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Weslaco, Texas

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G. O. Niemann, J. M. Miller, J. Jenkins, L. R. Withers and W. L. Bryan
Florida Citrus Research Foundation
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POTENTIAL OF PROCESSED PRODUCTS FROM MUSCADINE GRAPES

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Essence and concentrate were prepared from cold-pressed and detartrated juices from two widely grown cultivars of Muscadine grapes (3) and evaluated in cooperative research with the University of Georgia: 1) Higgins, a large bronze variety noted for high yield of amber juice, which is also becoming popular in Florida (4), and 2) Hunt, a black cultivar with deep red juice. About 50 gallons of juice from each variety were steam stripped at atmospheric pressure to recover essence containing some of the most volatile flavor components. The cooled, stripped juices were then concentrated five-fold using a pilot TASTE evaporator (Florida Department of Citrus, Lake Alfred) to yield 65°Brix products. Essences are being analyzed to identify major flavor compounds and flavor effects are being evaluated in products.

Some properties of fresh juice and reconstituted concentrate are shown in Table 1. Sugar content was about 13%, titratable acidity about 0.4-0.5% (tartaric acid) and pH 3.0-3.2. The °Brix/acid (°B/A) ratio of reconstituted concentrate was higher than for fresh juice, probably because more potassium acid tartrate had precipitated. Reconstituted Hunt concentrate had less red color than fresh juice (lower absorbance at 520 nm), probably from loss of anthocyanin pigments during thermal treatment (1). Concentrate from the bronze variety reconstituted to a darker yellow color than fresh juice, similar to the effect of hot pressing compared to cold pressing (2).

Table 1. Muscadine juice and reconstituted concentrate.

	°Brix	pH	°B/A	Absorbance* 420nm	Absorbance* 520nm
Hunt (red)					
Fresh juice	12.8	3.0	24	0.17	0.63
Recon. conc.	12.7	3.0	25	0.15	0.53
Higgins (amber)					
Fresh juice	12.6	3.2	30	0.29	-
Recon. conc.	12.4	3.2	33	0.49	-

*Diluted 1:10 for light absorbance.

Aqueous essences from both juices contained the major components listed in Table 2, but in different amounts. These were determined by extracting essence with Freon 11 or methylene chloride, removing most of the solvent by vacuum distillation and analyzing the residue using gas chromatography, infrared and mas spectroscopy. Odor was evaluated from each essence component separated by gc, and β-phenylethanol was judged highly important

to Muscadine odor, although many other components had fruit-like aromas that probably contributed to Muscadine flavor.

Table 2. Major Muscadine essence components.

Alcohols	Esters
Methanol	Ethyl acetate
Ethanal	Ethyl propionate
Butanol	Propyl acetate
2-Methyl-butanol	Butyl acetate
Hexanol	Benzyl acetate
trans-2-Hexen-1-ol	Ethyl cuprate
β-Phenylethanol	
	Hydrocarbons
Aldehydes	Toluene*
Acetaldehyde	m-Xylene*
trans-2-Hexenal	d-Limonene

*Toluene and xylene may be components of extracting solvent.

Reconstituted concentrate was compared with fresh juice (from frozen grapes) by a 12-member panel familiar with Muscadine products using Hedonic ratings (Table 3). Color scores of reconstituted concentrates were acceptable but lower than those of fresh juice. Aroma and flavor of reconstituted concentrates were significantly preferred over fresh juices. However, Higgins essence was added at a 3x higher level than originally present in fruit juice. Jellies and wines have also been prepared and are under evaluation, as well as blends of Muscadine juices with other juices including citrus. Aroma and flavor of jellies prepared from concentrates with essence were also preferred over those from fresh juice, while these jellies were not significantly different in color scores. These preliminary studies show the promising new product potential of processed Muscadine grape juice and concentrate from the Southeastern U.S. and Florida.

Table 3. Hedonic evaluation of juices (1-9 scale).

	Color	Aroma	Flavor
Hunt			
Juice from frozen grapes	8.5	6.2	6.0
Recon. conc. + essence (1x)	7.0	7.7	7.3
Higgins			
Juice from frozen grapes	7.3	7.1	6.4
Recon. conc. + essence (3x)	6.4	7.5	7.5

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VOLATILE FLAVOR CONSTITUENTS OF FLORIDA GUAVA

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Guavas are used commercially mainly in the preparation of jams, jellies and mixed tropical fruit drinks. Although they are not grown extensively in the Continental United States, plantings are found in Hawaii and other tropical areas throughout the world. Even though guavas make a desirable flavor contribution to processed food products, the chemical composition of guava fruit has received relatively little attention. In the only systematic study on flavor components of guava, Stevens et al. (1) identified 22 components of a volatile fraction from guava puree.

We have recently identified 21 constituents, including 12 components not previously reported in guava, and studied the significance of certain oxygenated constituents to guava flavor. A puree obtained from wild Florida guavas was extracted with CH_2Cl_2 to obtain a dark viscous oil with a characteristic aroma of fresh guava. This solvent extract was separated by preparative thin-layer chromatography (TLC) into four fractions. Each fraction was analyzed by gas chromatography-mass spectrometry (GLC-MS) combination, by IR analysis of samples from analytical GLC separations of the TLC fractions, and by comparison of GLC retention times with authentic samples.

The identified compounds are listed in Table 1. Most of the guava aroma was in the TLC fractions of intermediate polarity (2 and 3 of Table 1) and cinnamaldehyde, cinnamyl acetate, and methyl cinnamate probably contribute the most to the overall guava aroma, with cinnamyl acetate being the most important contributor of the three to characteristic fresh guava aroma.

Table 1. Identified components from TLC fractions of guava puree extract.

TLC Fraction 1	TLC Fraction 3
Hydrocarbons	Esters
β-Pinene*	Cinnamyl acetate
Limonene	Methyl cinnamate
β-Copaene*	Methyl butyrate
β-Caryophyllene	Phenyl ethyl acetate
Farnesene*	
α- & β-Humulene*	
β-Bisabolene	TLC Fraction 4
β-Selinene*	Alcohols
α-Selinene*	
Δ-Cadinene*	Cinnamyl alcohol*
Curcumene*	Phenyl ethyl alcohol
	Nerolidol*
TLC Fraction 2	
Aldehydes and Ketones	
Cinnamaldehyde*	
Benzaldehyde	
β-Ionone	

*Newly reported guava constituent.
Fractions one (hydrocarbons) and four (alcohols) did not contribute significantly to the guava aroma.

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TROPICAL SEED OILS AS REPLACEMENTS FOR COCOA BUTTER

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Substitutes for cocoa butter have been sought for over two decades because of the increasing high cost of this product. These substitutes have included synthetics [Coberine and Nucoa S (2)], hydrogenated vegetable oils [e.g. cottonseed (2)], and fractions from the crystallization of beef tallow (3).

Cocoa butter retains its value in the confectionery market because of its unique melting properties. Primarily, the melting range of an oil is dependent upon its fatty acid composition and the distribution of fatty acids connected by ester linkages to the glycerol moiety. In cocoa butter, two saturated acids, palmitic (P) and stearic (S), and one monounsaturated acid, oleic (O), comprise over 95% of the fatty acids. About 70% of the distributed acids are found in a disaturated combination, e.g. POP, POS and SOS.

Representative plants from 13 tropical families were screened for their seed fatty acid contents. These included: Sterculiaceae (cocoa butter and Java olive), Rutaceae (Valencia orange and Duncan grapefruit), Rosaceae (peach), Lauraceae (avocado), Myrtaceae (guava), Combretaceae (tropical almond), Guttiferae (Gamboge tree), Ebenaceae (velvet apple), Sapindaceae (lychee and logan-3 cultivars), Annonaceae (sugar apple), Bombacaceae (South American sapote), Sapotaceae (Mamey sapote) and Anacardiaceae (Brazilian pepper and mango-4 cultivars).

Oils were obtained from ground seeds by extraction with hexane at room temperature. Fatty acid methyl esters (FAME's) were prepared by transmethylation with BCl_3, HCl or $NaOCH_3$. Oils from four samples were fractionated according to their degree of unsaturation by silver nitrate TLC and the FAME's prepared from these different silver nitrate separated zones. Gas chromatographic analyses (Table 1) showed only mangoes and the Mamey sapote to have fatty acid profiles similar to cocoa butter. Silver nitrate triglyceride analyses showed Keitt mango to be similar to cocoa butter. In Haden mango seed, slight change occurred during fruit ripening. This is in contrast to the marked changes that occur in the flesh of the fruit.

Table 1. Major fatty acids in seed oils.

	Fatty Acids			
Oil	Palmitic 16:0	Stearic 18:0	Oleic 18:1	Other
1. Cocoa butter *Theobroma*	25.5	32.9	38.4	3.2
2. Java olive	22.7	2.0	9.1	66.2*
3. Valencia orange	27.3	4.4	28.3	40.0
4. Duncan grapefruit	30.0	3.6	21.6	44.8
5. Peach	7.4	1.9	61.9	28.8
6. Avocado	23.8	1.8	14.6	59.8
7. Guava	11.0	3.9	11.0	74.1
8. Tropical almond	16.9	5.6	25.6	51.9
9. Gamboge tree	28.0	8.8	43.2	20.0
10. Velvet apple	25.2	2.4	43.1	29.3
11. Lychee	14.3	4.5	30.5	50.7
12. Logans (3)	17.2	7.9	38.5	36.4
13. Sugar apple	19.5	5.7	21.0	53.8
14. So. Am. Sapote	22.3	12.5	29.8	35.4
15. Mamey Sapote	10.1	21.1	58.0	10.7
16. Brazillian pepper	19.4	6.4	39.9	34.3
17. Mangoes (4)	7.4	40.8	44.9	6.9

*Contains 56.9% cyclopropane fatty acids.

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DETERMINATION OF PHTHALIDES IN CELERY SEED OIL

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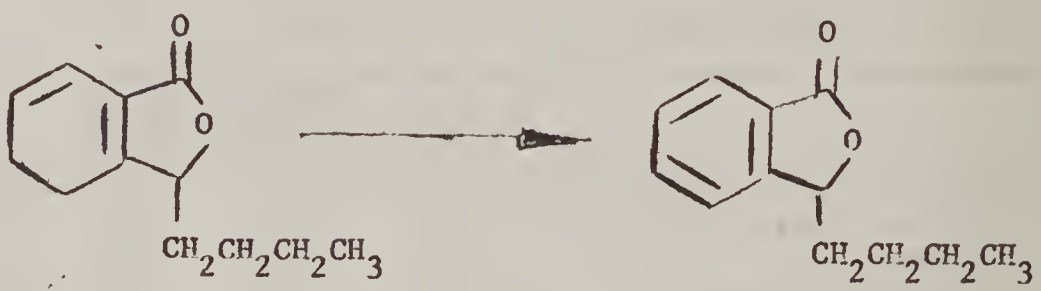
Some phthalides (C_{12} lactones) are thermally unstable and difficult to determine by gas chromatography (GC). Celery seed oil contains sedanenolide (sedanonic anhydride) 1 and 3-n-butyl phthalide 2 as the major phthalide components. Compound 1 was unstable under our GC conditions and was converted to 3-n-butyl phthalide along with smaller amounts of a number of other phthalides.



1

λmax 225, 276 nm

E_{254} 8.0

2

λmax 238, 275, 282 nm

E_{254} 1.9

Figure 1. Celery seed oil phthalides.

A more accurate determination of these important celery flavoring compounds can be obtained by liquid chromatography. Conditions were found for quantitation of the major phthalides in celery seed oil or seed extract by both TLC and HPLC.

TLC

Silica gel GF plates developed with benzene-1% ethanol separated the phthalides in seed oil or seed extract. Because of the strong UV absorption (Fig. 1) spots could be readily visualized by UV illumination. Anisaldehyde-phosphomolybdic acid spray reagent also proved useful (2). Phthalides appeared as dark spots under 254 nm illumination at R_f 0.3-0.6 (air-dried plates).

On preparative plates the phthalides from two commercial distilled seed oils (A and B) and a seed extract were separated and visualized using Rhodamine B spray and 366 nm illumination from beneath the plate. The bands were eluted, checked for purity by IR, and weighed for quantitative analysis (Table 1).

HPLC

On a polar, bonded phase (Durapak-Carbowax 400, hexane + 0.1% isopropanol), the phthalides were separated cleanly from seed oil, but not seed extract. The latter contained a large lipid (triglyceride) fraction which eluted at the same time as sedanenolide and interferred with analysis. Silica (Porasil) separated the two phthalides, but both were contaminated with an unknown impurity which eluted along with them. Reverse phase (C_{18}-Corasil) did not separate the compounds adequately. A UV detector (254 or 280 nm) detected eluting peaks very well because of the strong UV absorption of both compounds. Peaks were collected from a preparative Carbowax column and IR spectra were obtained for purity verification. The quantitative analysis shown in Table 1 was obtained from values for peak areas relative to those of 3-n-butyl phthalide standard solutions.

As Figure 2 shows, 3-n-butyl phthalide 2 elutes from Carbowax as a sharp peak just before sedanenolide 1 and more reliably serves as a standard. Although the butyl phthalide peak looks relatively small, the relative amount is much larger than indicated from the peak areas shown because of the 4-fold difference in extinction coefficients (Figure 1).

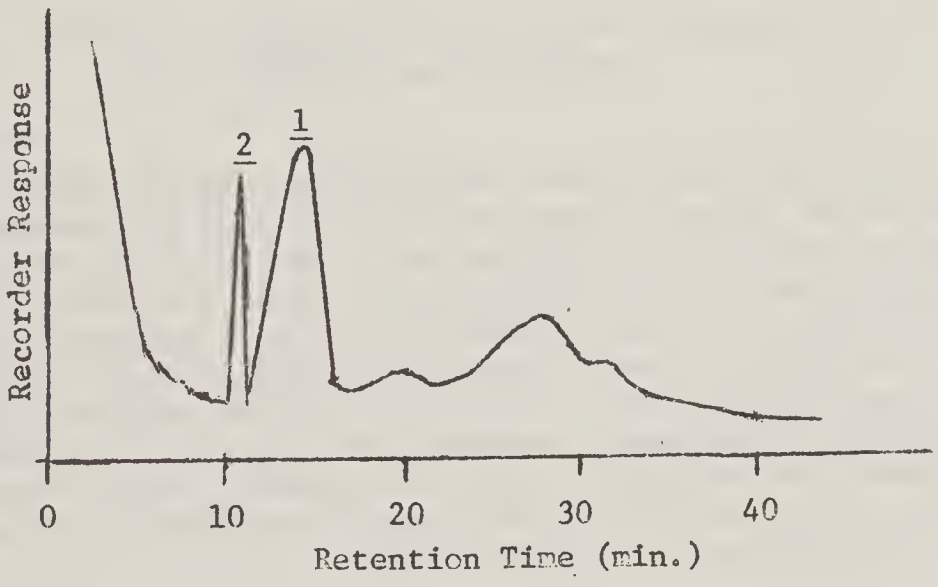


Figure 2. HPLC trace of seed oil B.

Comparison of Methods

Values for the two methods (Table 1) are comparable within the estimated error of ± 10%. The GC method was consistently low. The TLC techniques with some type of UV assay would probably be the best method for both oils and extracts. Although this technique requires about twice the time as HPLC (1-1.5 hr.) 10 to 20 samples could be separated simultaneously on a large plate.

For rapid analysis of distilled oil samples, HPLC would be the best method. Analysis time could probably be shortened further by optimizing column packing and flow rate.

Table 1. Celery oil compositions (wt. %)

	Oil A		Oil B		Seed Ext	
Method	2	1	2	1	2	1
TLC	3.6	9.5	2.8	3.7	2.1	7.4
HPLC	4.4	8.8	3.2	4.0	-	-
GC	3.4	3.6	1.4	1.3	0.4	0.7

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SOLAR APPLICATIONS IN FOOD PROCESSING: A NATIONAL ERDA/ARS RESEARCH PROGRAM

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The Energy Research and Development Administration (ERDA) has been developing, over the past several years, a broad effort to encourage increased research and development on processes maximizing use of solar energy and thereby conserving conventional fossil fuel sources. Funds for this program are provided by ERDA as part of the overall objective of reducing U.S. dependence on natural gas and imported energy supplies. These funds are channeled through ARS, USDA, to provide nationwide coordination of the research effort, to provide agricultural expertise in managing the program and to make optimum use of the existing ARS technical management system. The Cooperative Research Service (CSRS) through the State Agricultural Experiment Stations (SAES) assists with program planning and management.

Table 1. ERDA/USDA Research Programs on Agricultural Applications of Solar Energy.

Program	Contact	Year
Grain Drying	G. G. Hartsock, 2336 Northwestern Avenue, West LaFayette, Indiana 47906	3rd
Heating/Cooling of Greenhouses and Rural Residences	T. E. Bond, Box 792, Clemson, South Carolina 29631	3rd
Drying Field Crops Other Than Grain	J. L. Butler, Georgia Coastal Plain Experiment Sta., Tifton, Georgia 31794	2nd
Animal Production and Livestock Shelters	Floyd Reese, P. O. Box 5367, Mississippi State, Mississippi 37962	2nd
Food Processing	R. E. Berry, P. O. Box 1909, Winter Haven, Florida 33880	1st

Table 1 indicates the five general research programs devoted to solar energy applications in agriculture, now underway. The contact person listed in this table for each program is the Principal Investigator and Coordinator of that program for ARS. Two programs are in their third year, two in their second year and one has just been initiated. A brief description will be given of several specific projects under the first four listed programs. However, the principal description will be of the most recent program, i.e., Food Processing, since it is in its first year and most of the projects there have not been described or publicized to any significant extent.

The broad objective of the Food Processing Research Program is to determine methods of using solar energy with emphasis on applications, development and demonstration of feasible systems with significant portions of the energy derived directly from solar energy. Some background projects on feasibility and compatibility are studying energy demands of certain food processing operations and the potential of direct solar energy for heat supply, using computer modeling and broad surveys. Additional support of research in these areas will be minimal. Also, because relatively conventional applications of solar heated water and air are covered in other ERDA research programs proposals of this nature are not encouraged. That is, the program will not support studies of normal uses of hot water or hot air where the water or air is simply provided from a solar heater. Rather, proposals of unconventional product or process development to adapt food processes to more effectively or more widely use solar energy sources are encouraged. Creative and innovative approaches to using direct solar energy in food processes and on products are also encouraged. It is anticipated that this research program can lead to significant changes in food processes in general, to enable larger percentages of total energy from those processes to be derived from solar sources. It is also anticipated that new, unconventional approaches to food processes will be developed, which can become economically attractive in the future as conventional fuel sources increase in cost.

Principal responsible individuals involved in managing this program are:

Project Coordinator for ERDA:
Mr. William R. Cherry
Division of Solar Energy
Washington, D.C. 20545

Principal Investigator for ARS Food Processing Program:
Dr. R. E. Berry
P. O. Box 1909
Winter Haven, Florida 33880

Project Coordinator for the Agricultural Research Service:
Dr. Landy B. Altman
Room 219-North Building
Agricultural Research Center West
Beltsville, Md. 20706

Co-Principal Investigators for SAES, Food Processing Program:
Dr. Fred H. Buelow, Chairman
Agricultural Engineering

and

Dr. Daryl B. Lund
Food Science Department
University of Wisconsin
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Titles and brief abstracts of on-going projects currently supported in this program are listed below:

Michigan State University, East Lansing
Dr. F. W. Bakker-Arkema - Project Manager
Feasibility of Solar Water Heating in Food Processing in the Mid-Western U.S.

An investigation into feasibility, fossil energy saving potential and economics of using solar heated water in food processing plants in the mid-west. First year's study includes surveys of meat, milk, and fruit-vegetable areas.

University of Wisconsin, Madison
Dr. F. H. Buelow and Dr. Daryl B. Lund, Project Managers
Compatibility of Solar Energy Supply, Collection and Storage with Food Processing Energy Demands

Investigation of compatibility of solar energy systems with food processing systems. Hot water and steam demand of selected food processes will be determined. Computer models will be developed to use as input parameters in the solar energy model at the Univ. of Wisconsin Solar Energy Lab.

Colorado State University, Ft. Collins
Dr. Charles C. Smith, Project Manager
Development and Demonstration of Solar Process Drying of Potato Products

An optimized solar dryer for potato products will be designed, a prototype fabricated and operated, to determine energy and production efficiency and general quality of products.

Oklahoma State University, Stillwater
Dr. G. H. Brusewitz, Project Manager
Solar Processing Waste From Meat Packing Plants

Pilot size solar still will be constructed for drying beef packing plant paunch (slaughtered animal stomach and intestinal contents) as a meat processing plant by-product. Potential energy savings will be determined and portable experimental solar stills will be tested to determine operating parameters.

University of Hawaii, Honolulu
Dr. J. H. Moy, Project Manager
Air Drying, Freeze-Drying and Osmovac Dehydration of Foods With Solar Energy

Current conventional food processes of air drying and freeze-drying will be adapted to enable incorporation of more low quality energy and produce acceptable dry fruit and vegetable products. Osmovac dehydration will be used to concentrate fruits and juices, with solar energy to concentrate osmotic pressure solutions. Reflectorized dryers will be used to dry tropical root crops.

U. S. Citrus and Subtropical Products Laboratory
Dr. W. L. Bryan and Dr. R. E. Berry, Project Managers
Dehydration of Southeastern Fruits and Vegetables by Solar Energy

A practical process will be developed for drying southeastern fruits and vegetables using optimal amounts of concentrated solar energy as principal energy source, augmented by fossil energy sources as needed. A batch-type solar dehydrator will be designed and constructed, allowing up to 3x solar energy on the food product. This will be tested on traditionally dried as well as unique fruits and vegetables.

POTENTIAL OF SOLAR ENERGY FOR FOOD DEHYDRATION

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Because of current trends toward higher cost of fossil fuels and uncertainty regarding the future, solar energy probably will become economically feasible for food dehydration. Several methods of recovering and using solar energy are technically feasible. However, because of inherent solar limitations, initial equipment investment would be higher than for conventional drying:

1) Large-area collectors would be required because of the diffuse nature of solar radiation (average solar energy, Central Florida: 12,000 B/m^2-day in December - 22,000 B/m^2-day in June).
2) For continuous operation, solar energy must be collected daily (8-10 hrs) and stored for overnight use.
3) Commercial applications would require a standby conventional heating method to insure continuous operation during extended overcast weather.

Many dried fruits and vegetables are commercially produced in the Western states, some by direct solar drying but most use enclosed hot-air dryers heated by natural gas. Development of new solar processes would reduce use of natural gas or alternate fuels and could lead to new industries in other agricultural areas with abundant sunshine, such as the Southeastern U.S. Central Florida, for example, receives an annual average of 6.0×10^6 B/m^2, enough to evaporate 2.8 tons/m^2 of water. Some future applications of solar drying in the citrus industry might include reducing fuel required for drying citrus pulp and peel for cattle feed or for drying orange juice sacs for drinks.

Our project objectives are: 1) to develop dehydration processes which are economically feasible and compatible with solar limitations, and 2) to

demonstrate acceptable products prepared by these processes. Initially, we are developing a 24-hour drying process using: stage 1 - 6-8 hr direct and reflected solar energy to remove maximum moisture during the initial high-rate drying period, followed by stage 2 - low-temperature air overnight to complete drying, when final drying rate is controlled more by internal diffusion of moisture than by surface heat transfer. A solar dehydrator constructed by the Florida Solar Energy Center for these studies (1), contains two enclosed drying compartments with double glazing (Thermopane glass) above, and beneath food trays constructed of corrugated stainless steel mesh (0.30 m^2/tray). Two adjustable flat reflectors are pivot-mounted, one above and behind and one underneath to concentrate radiation 1-2x on the food trays. Entering air rate, temperature and humidity can be controlled and flow directed upward or downward through the trays.

Studies underway will define drying conditions using maximum solar energy (without overheating the product) and minimum auxiliary hot air needed to prepare products with acceptable moisture. Preliminary tests have been conducted with vegetables (diced carrots and green peppers, celery and parsley leaf), mango slices, peach halves and orange juice sacs. Dried products compared favorably with reference samples prepared by conventional hot-air drying (2).

Dryer operation was characterized in 4 tests with diced carrots (1/2" cubes) with an air rate of 50 cfm during both drying stages. Ambient air (about 96°F, 45% RH) was used for stage 1, while ambient air was heated to about 110°F (37% RH) during the 18-hr stage 2 air drying. Table 1 shows % water evaporated during each stage while operating with different amounts of direct and reflected solar radiation. Thus, with ambient air only (test 1 - no direct or reflected radiation), more than 1/3 of the water in product was evaporated during stage 1, and more than 1/2 was evaporated using heated air during stage 2. With addition of solar radiation in tests 2-4, more water was evaporated during stage 1, reaching about 3/4 with both reflectors (test 4), leaving less water to be evaporated in stage 2.

Table 1. Drying results for diced carrots.*

Test no.	Conditions	Water evaporated, % 1st Stage	2nd Stage	Total	Solar Efficiency**
1	No solar	35	55	90	0
2	Direct solar only	60	35	95	45
3	Direct + top reflector	64	31	95	52
4	Direct + both reflectors	74	22	95	66

*1/2" cubes, initial moisture = 91%, tray loading = 11.1 kg/m^2.
**Based on direct solar radiation to a horizontal surface, 0.30 m^2.

Solar efficiencies were estimated from additional evaporation beyond that with ambient air (test 1), based on daily direct radiation to the food tray (measured by an Eppley pyranometer). Thus, in test 2 45% of direct rediation

on the glass cover was transmitted and absorbed by product to cause additional evaporation. Addition of both reflectors increased efficiency on this basis to 66%. Shrinkage of product late in stage 1 tended to reduce solar efficiency because the tray surface was not entirely covered and all available radiation was not used.

Practical applications of solar energy to food drying could vary from small-size home operations to large-scale commercial installations, each with design factors depending upon capacity and operational restrictions. A small solar dryer for home or community might use both direct and reflected radiation and be used only during daylight hours, thus extending drying time beyond 1 day for some products. A commercial installation would more likely be operated on a 24-hour basis, using stored solar heat overnight, with auxiliary conventional heat as required. For a commercial dryer, the land area required for adequate collectors might become a limiting factor. Planned studies will help determine size limitations of installations for different practical applications of solar energy in food drying.

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NEW COMPOUNDS IN OILS FROM ABSCISSION TREATED ORANGES

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The rind-injuring abscission agents we have investigated affect a particular metabolic pathway within the orange, regardless of cultivar or maturity, causing the production of the same group of phenolic ethers. All abscission agents that have yielded successful results on oranges before mechanical harvesting are of the rind-injuring type. For the current study, product samples were prepared from barely-, and well-matured Hamlin, Pineapple and Valencia oranges treated with one of the promising citrus abscission agents (Acti Aid, Release, Pik-Off, Sweep) and compared with samples from untreated control products from oranges picked from adjacent trees. A detailed compositional analysis of the cold-pressed peel oils revealed the presence of a series of phenolic ethers found for the first time in citrus products - but only in abscission-treated samples. The taste threshold of each new compound was determined by the method described by Harrison and Elder (1). Each taster was presented with a series of paired samples, randomly arranged. Each pair consisted of one dilution of the experimental compound and an untreated control sample. The threshold level was the concentration correctly identified by 75% of the panel.

In order to determine whether the flavor contributions of the newly found phenolic ethers were additive, three of these compounds were added to orange juice at one half their threshold levels and compared to a control sample. The panel significantly distinguished the experimental sample at the 99.9% confidence level. The structure and name of each new compound and flavor threshold in orange juice of some of the compounds, are shown in Figure 1.

The concentration of the most potent of these, eugenol, was determined to be 239 ppm in peel oil and 20.8 ppb in single-strength juice from abscission treated oranges. The concentration in juice was calculated on the basis of a 0.0175% level of peel oil present in the juice. Although 20.8 ppb eugenol is slightly below the threshold concentration, the additive flavor effect demonstrated by these ethers shows that the level of eugenol present probably is high enough to influence flavor quality in both single-strength juice or pumpout juice flavored with experimental oil.

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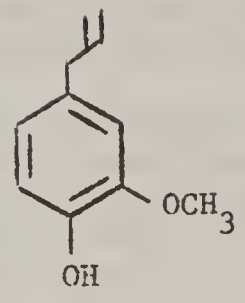
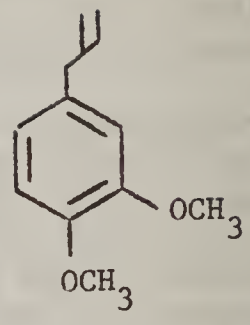
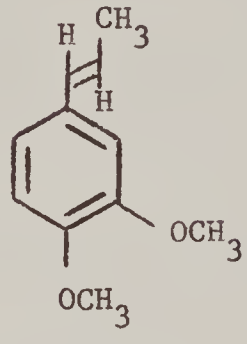


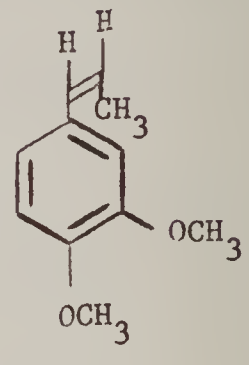
Eugenol
Flavor threshold in OJ - 22 ppb



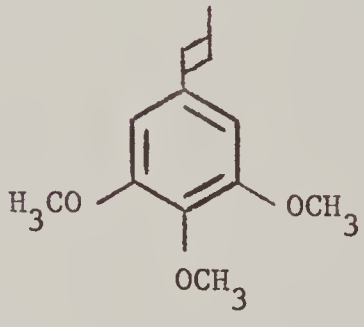
Methyl eugenol
Flavor threshold in OJ - 1.25 ppm



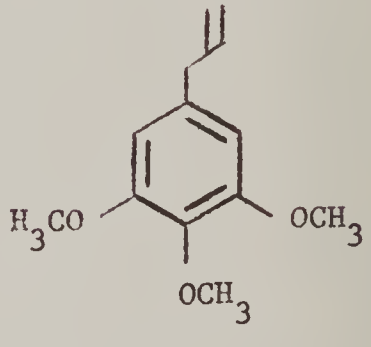
trans-Methyl
iso-Eugenol
Flavor threshold in OJ - 35 ppm



cis-Methyl
iso-Eugenol
Flavor threshold in OJ not determin



iso-Elemicin
Flavor threshold in OJ not determined



Elemicin
Flavor threshold in OJ - 22 ppm

Figure 1. New compounds in oils from abscission-treated oranges.

RECENT STUDIES OF LIMONOID BIOCHEMISTRY

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The problem of limonin bitterness of citrus juices and other processed products is becoming more widely recognized each year. We are currently dealing with this problem by means of two approaches; one is an enzymic debittering process and the other, which was initiated recently, is a preharvest treatment of citrus to reduce the limonoid content of fruit tissues. Recently we found that citrus leaves, particularly young ones, are the active site of the biosynthesis of limonoids. Radioactive tracer work showed those limonoids synthesized in leaves are translocated to fruit tissues. Limonoids accumulated in seeds are also synthesized in leaves and translocated to the seeds. No evidence was found of limonoid synthetic systems in seeds.

Derivatives of triethylamines such as 2-(4-chlorophenylthio)triethylamine chloride and many others have been shown to inhibit the cyclase(s) in carotenogenesis in citrus and microorganisms. Since limonoids are cyclic terpenoids, similar cyclases may also be involved in biogenesis of limonoids. If so, such triethylamine derivatives might inhibit formation of limonoids in citrus. To test this we chose two compounds, 2-(4-ethylphenoxy)triethylamine and 2-(3,4-dimethylphenoxy)triethylamine to study effects on biosynthesis and biodegradation of limonoids in lemon leaves. Both compounds markedly inhibited biosynthesis of limonoate A-ring lactone (LARL) in lemon leaves. Eight days from treatment, leaves sprayed with 300 or 500 ppm of 2-(4-ethylphenoxy)-triethylamine, respectively, contained 1/3 or 1/12 as much LARL as control leaves. Compound 2-(3,4-dimethylphenoxy)triethylamine was less effective, but LARL content was significantly lower than that of the control. After 6 and 10 days treatment, control leaves contained about 2.5 times as much LARL as leaves sprayed with 300 ppm. Radioactive tracer work showed, however, that neither compound appeared to affect the biodegradation of limonoids in leaves. Results to date strongly suggest preharvest application of triethylamine derivatives to citrus leaves at early stages of fruit growth may offer a new approach to controlling limonoid content of citrus.

Our studies of bacterial limonoid metabolism have shown limonoids are metabolized in bacteria by two pathways; one through 17-dehydrolimonoids and the other through deoxylimonoids. The former pathway has been shown to be present in citrus. Preliminary studies indicated that the latter is also present in citrus. Several enzymes involved in each pathway have been isolated and characterized. The possible application of bacterial enzymes to debitter citrus juices will also be discussed.

PECTINASE IN THE PREPARATION OF GRAPEFRUIT FOR SECTIONIZING

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Grapefruit is processed commercially for salad sections by a procedure that requires hot water and hot caustic to remove the peel and adhering membrane (Figure 1). The peel is softened by holding the fruit in bins for 4 to 6 days before sectionizing. The fruit are then treated with scalding hot water (91 to 99°F) for 5 to 8 minutes before they are conveyed to the mechanical peeler. From the peeler the fruit are conveyed to the hot caustic bath for finishing. After 12 to 25 seconds in the caustic and 15 to 25 seconds for draining, the fruit are rinsed, then chilled, before moving to the sectionizer. Although cold water for cooling is recycled, the rinse water is not. The hot caustic solution is recycled until the fruit solids build up when the solution is discarded. The waste caustic solution presents a disposal problem in biological waste treatment systems that operate with low flow-through volumes.

We have investigated effectiveness of pectinase solution in removing peel and preparing fruit for sectioning. The essential step in the pectinase procedure is vacuum infusion of pectinase solution into scored intact or peeled grapefruit (Figure 1). Vacuum infusion resulted in saturation of the albedo with pectinase solution. When the solution contained 400 ppm pectinase, the peel could be removed in 10 minutes (Table 1). At lower concentrations of enzyme additional time was required. The time-temperature-concentration requirements for removing peel from intact fruit was the same as for removing the residual albedo and membrane from hot water peeled fruit. The solution of 400 ppm pectinase slowly lost activity at 50°C so the infusion bath was kept at 30°C and the fruit was warmed to 60°C before infusion and held at 50°C afterwards. Sections were easily excised from pectinase treated grapefruit compared to untreated grapefruit. Sections prepared from enzyme treated fruit were less bitter than those prepared by hand. Peel juice from pectinase-treated grapefruit contained about 7 g naringin, 0.2 gm pectic acids and 0.8 gm water soluble pectin per liter. These amounts vary with concentration of pectinase and treatment time. If these substances could be recovered economically from the peel they would be useful by-products with established markets.

Enzymic treatment to prepare grapefruit for sectioning has several advantages over the conventional procedure which requires 1) 4-6 day storage, 2) cure, 3) scalding water and 4) caustic wash. The major operational benefits of developing an enzymic system are elimination of the cure/storage requirement and the caustic disposal problem, and lower water consumption. Another benefit is quality improvement. The storage cure increases anaerobic metabolism and accumulation of fermentation products, many of which produce off-flavors in the fruit. The scalding hot water treatment to soften the peel and subsequent caustic finish also may impart a cooked off-flavor to the sections. Pectinase infusion would eliminate the need for these practices and thus a source of off-flavors.

Table 1. Pectinase concentration for peel removal at 50°C.

Pectinase mg/l	Holding time min
400	10
200	20
100	30

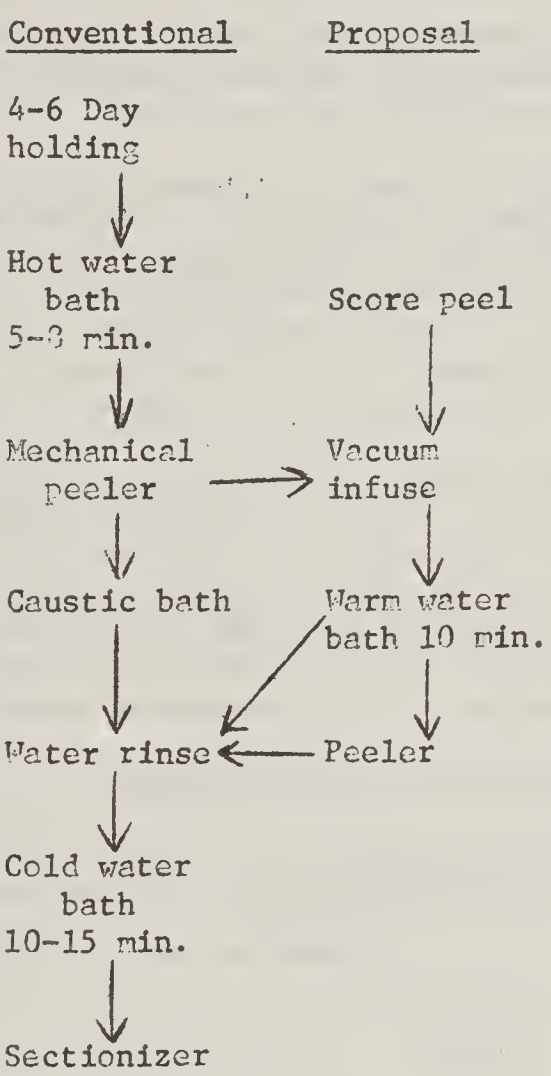

Figure 1. Conventional and proposed procedure to prepare grapefruit for sectionizing.

FLAVOR CHANGES OF ESSENCE-FORTIFIED FCOJ DURING FROZEN STORAGE

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Although essence and essence oil are widely used to impart 'fresh juice' flavor to FCOJ, the flavor enhancement may diminish before the product reaches the consumer, particularly under improper storage conditions. Studies by Guadagni et al., (2) showed a taste stability of 62 weeks at 0°F for FCOJ with aroma solution, while Dougherty et al., (1) showed 30 month stabilities at -2°F for concentrates fortified with three different aqueous essences. There are no reported studies on essence fortified Florida FCOJ stored at temperatures of 0-32°F. Flavor fortified FCOJ may experience such temperatures after processing and before final consumer use, either in transit, in the store, or in the home. Preliminary studies to evaluate possible changes in effectiveness of essence in FCOJ above 0°F are in progress.

These flavor studies include: 1) storage times which cause significant detectable flavor differences and significant preferences, and 2) preferences between the different concentrates after selected storage times. Table 1 identifies type and amount of flavor fortification used for experimental concentrates, and results from initial flavor tests. All samples were prepared from a 65°Brix concentrate diluted to 45°Brix. Storage temperatures for six-ounce cans of each sample included -120, 0 and 20°F.

Detectable flavor differences shown in Table 1 were established by trained panelists using triangular taste tests. Formulas 1, with cold pressed (C.P.) oil and 2, with C.P. oil and essence oil, were found different from 0°F stored controls after 3 weeks storage at 20°F. Formula 3 with aqueous essence added as well, was significantly different from its 0°F control after 1 week. These results tend to support reported observations that aqueous essence may lose effectiveness as a flavor enhancer at temperatures above 0°F.

Table 1. Storage stability of FCOJ samples.

	Flavor additives*			Time for detectable flavor change, wks**
Formula	C.P. oil %	Essence oil %	Essence %	20°F
1	0.018	0	0	3
2	0.012	0.006	0	3
3	0.012	0.006	1	1

*Additives as % in reconstituted juice.
**Triangular taste tests with trained panelists at confidence levels of 99% or better.

Present flavor evaluations using paired comparison tests with a panel of tasters who indicated a preference for essence in previous tests, have tentatively established storage times necessary for significant preferences. These times are within 1 to 2 weeks after detectable differences were found. This also further indicates the loss of effectiveness of aqueous essence in fortified concentrates, with storage. Preferences between the three stored concentrates are currently being studied.

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FEEDING STUDIES OF FREEZE-DRIED ORANGE PEEL PUREE AND FREEZE-DRIED ORANGE JUICE PREPARED BY SIMULATION OF THE WHOLE ORANGE PUREE PROCESS

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The nutritional value of citrus whole-fruit puree (1) is largely unknown due both to its limited traditional consumption among humans, and the relatively meager efforts to characterize its chemical composition.

Cattle feeding experiments (2) have established that long-term consumption of raw or dehydrated whole oranges or grapefruit was absolutely harmless to the animals. The studies reported here are the first on record to deal with non-ruminant animals.

Although there is no historical or scientific evidence to suggest that consumption of whole citrus or citrus peel may have detrimental effects, there is presumptive evidence in the literature which suggests that further research is needed to establish the effect of citrus peel and its components on the health of the consumer. The presumptive evidence consists mainly of various citations in the literature to known components of citrus peel which are biologically active and potentially antinutritive in character. It is the function of the research reported here to assess the merits of these presumptions by test feeding freeze-dried orange peel puree and freeze-dried orange juice.

On November 29, 1974 'Hamlin' oranges were harvested from a grove at the Texas Agricultural Experiment Station, Weslaco, Texas. The grove received documented care similar to that used in commercial orchards. At the Food Crops Utilization Research Laboratory whole oranges were water blanched as in the initial step of the whole orange puree process. After juice extraction, the peel and pulp underwent the remaining steps in the puree process of comminution, homogenization,

and pasteurization. The juice was also pasteurized. Both the puree and juice were freeze-dried. At the Western Regional Laboratory the freeze-dried products were fed separately at 30% of total diet to weanling rats for 45 and 90 days prior to necropsy. Body weights of the test and control animals were recorded at two-week intervals. Blood samples were collected at the time of necropsy for hematology and enzyme and chemical analyses. Organ weights, and gross pathological and histological examinations were made. The only adverse effect that was clearly evident at this high percentage of total diet was decreased body weight in the group fed the 30% freeze-dried orange peel. Some liver enlargement observed in this same group is most likely the result of microsomal stimulation which is not necessarily a toxic effect. Histological examination of various organs did not disclose any lesions which were associate with any of the three dietary groups.

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IMPROVED RECEIVING LINE FOR ORANGES AT PROCESSING PLANTS

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Luke R. Withers and William L. Bryan

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(Supported by The Florida Department of Citrus)
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A 25-box/minute pilot trash removal and grading system was installed and evaluated at Winter Garden C.P.C. in cooperative work with the Florida Citrus Research Foundation (funded by the Florida Department of Citrus), under direction of the U. S. Citrus and Subtropical Products Laboratory. The system with components listed below was installed to receive fruit from Winter Garden's Temple unloading line, and graded fruit were conveyed directly to processing without bin storage (Figure 1).

1) Bucket elevator (borrowed from Indian River Foods).
2) Cross-feed trash removal belt (4 ft x 8 ft), inclined 15° upward and tilted 10° downward in the direction of fruit flow (Figure 2).
3) Brush scrubber (52 in. x 8 brushes).
4) Mechanical grader (relocated from former pilot plant at Lykes Pasco Packing Company) that separated fruit into two streams by distance bounced from a 20 in. diameter rotating metal drum to a partitioned catch belt. Most cull fruit did not bounce as far as sound fruit and were concentrated into a cull stream for manual grading, while fruit which were projected over the partition were conveyed directly to processing. For mechanical grading, fruit

leaving the scrubber were aligned in 4 1/2 in. lanes on a roller conveyer laner (15° inclination) and on an acceleration ramp (5 ft. high, 65° inclination) before striking the bounce drum.

5) Roller conveyer (2 ft x 12 ft) for manually grading the cull stream from the mechanical separator before combining this stream with the good fruit stream to the plant.

Pilot system operability

The system was operated with both hand- and mechanically-harvested oranges intermittently throughout the season without breakdowns.

The inclined trash belt (Figure 2) separated all loose trash (leaves, twigs, etc.) and non-rolling cull fruit with negligible loss of good fruit. From several loads of mechanically harvested fruit, the trash system removed an average of 510 pounds/truckload (1240 pounds from one load), of which 80-90% was cull fruit. From hand-harvested oranges, an average of 200 pounds/truckload of trash was removed, and relatively little was cull fruit. The design of the trash belt was based on tests with a smaller model conducted at the USDA Laboratory.

Effectiveness of the reworked mechanical grader was improved, compared to last season, in part because essentially all trash had been removed from fruit before grading. However, fruit jamming between lane dividers, occasionally encountered last season with Valencias, was worse with smaller Hamlins. The lane dividers were removed from one side of the roller conveyer and essentially the same effectiveness of cull separation was observed when operating without lanes (Table 1). In both tests, more than half the culls were separated into a smaller stream (about 10% of total fruit) and concentrated about 5-fold compared with original fruit. Operating without lanes improved grading rate/unit width, while eliminating the problem of fruit plugging and reducing probability of mechanical breakdown.

Table 1. Results of operating with and without lanes.*

	Lanes	No lanes
Average fruit rate, boxes/min-ft width	5.0	6.0**
% Unwholesome in fruit load	4.7	4.6
% of total fruit separated by mechanical grader	10.7	9.4
% of total unwholesome fruit separated	52	51
% Unwholesome fruit in separated stream	23	25

*Mechanically harvested Valencia oranges.
**Instantaneous rates without lanes were as high as 10-12 boxes/min-ft width.

Unusual fruit conditions encountered during this freeze year included fruit with internal black rot, dried "puff balls" and very soft sound fruit. Of these:

1) Fruit with black rot could not be separated mechanically because they bounced the same as sound fruit.
2) "Puff balls" bounced considerably farther than sound fruit and the pilot system did not remove them, but could easily be modified to do so.
3) Some soft sound fruit were projected into the cull section, thus reducing efficiency of separating culls.

Controlled-rotation roller feeder for mechanical grading

When a conventional roller conveyor is operated at high linear speed required for a practical grading system, the rollers also rotate at high speed. This causes fruit to tumble end-over-end and reduces effectiveness of separating culls. An experimental "controlled-rotation" roller conveyer constructed at the USDA Laboratory could be operated to slowly rotate the fruit while conveying them at a high rate. With slow rotation, fruit rotated smoothly on their equators, which greatly improved uniformity of fruit orientation and trajectory to the grading ramp and drum.

Production systems

A production prototype trash removal and mechanical grading system has been designed for installation at Winter Garden CPC and evaluation over 3 seasons. This system will have two trash belts (one to remove trash and the other to remove some cull fruit) and a controlled-rotation roller feeder without a laning system. The 5-ft wide prototype grader is designed to separate 500 boxes of oranges in less than 15 minutes into two streams for manual grading of the smaller stream. The pilot system will remain intact at Winter Garden for limited production use, for evaluating methods to improve separation and for testing new concepts.

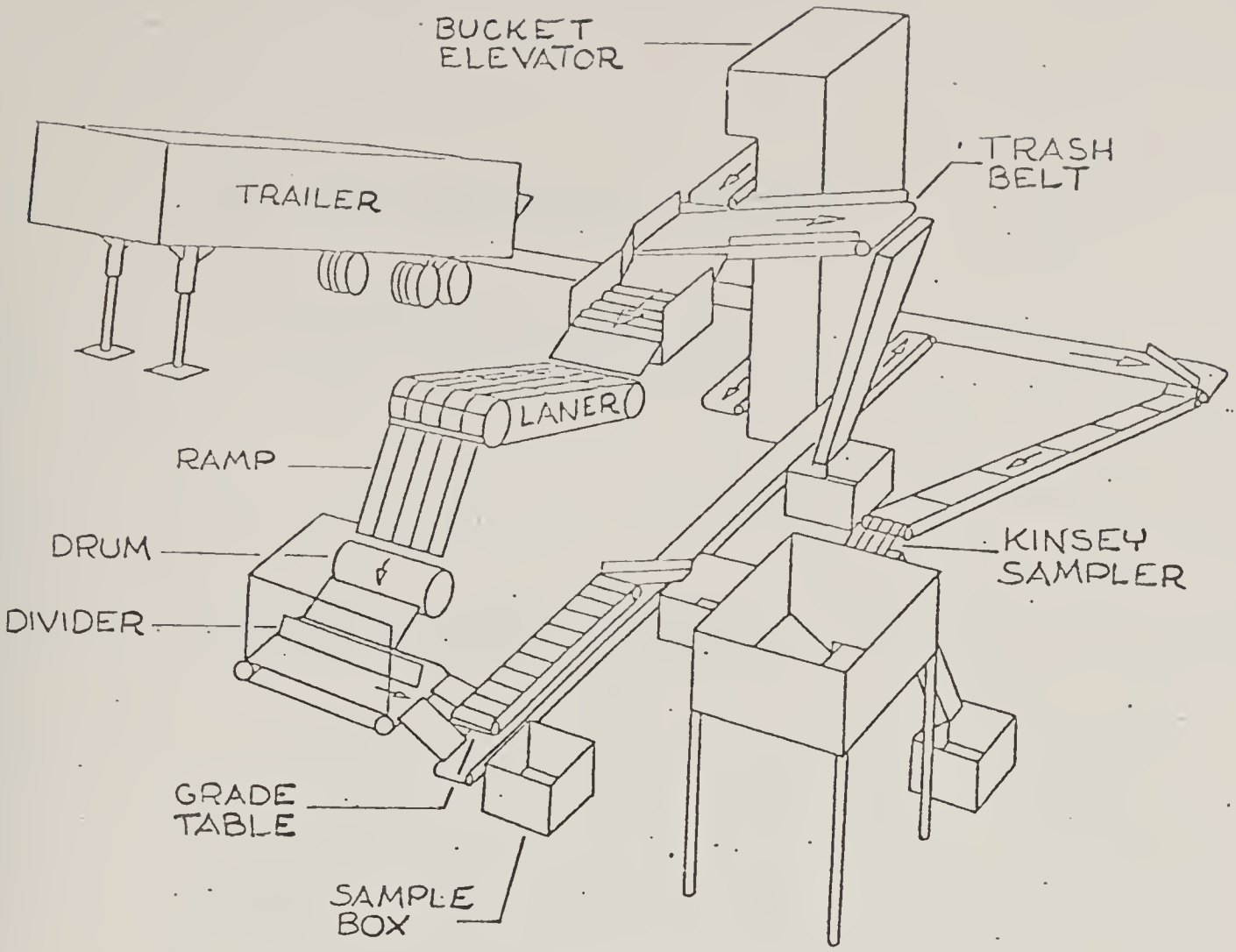

Figure 1. 25 Box/min pilot unloading line at Winter Garden C.P.C.

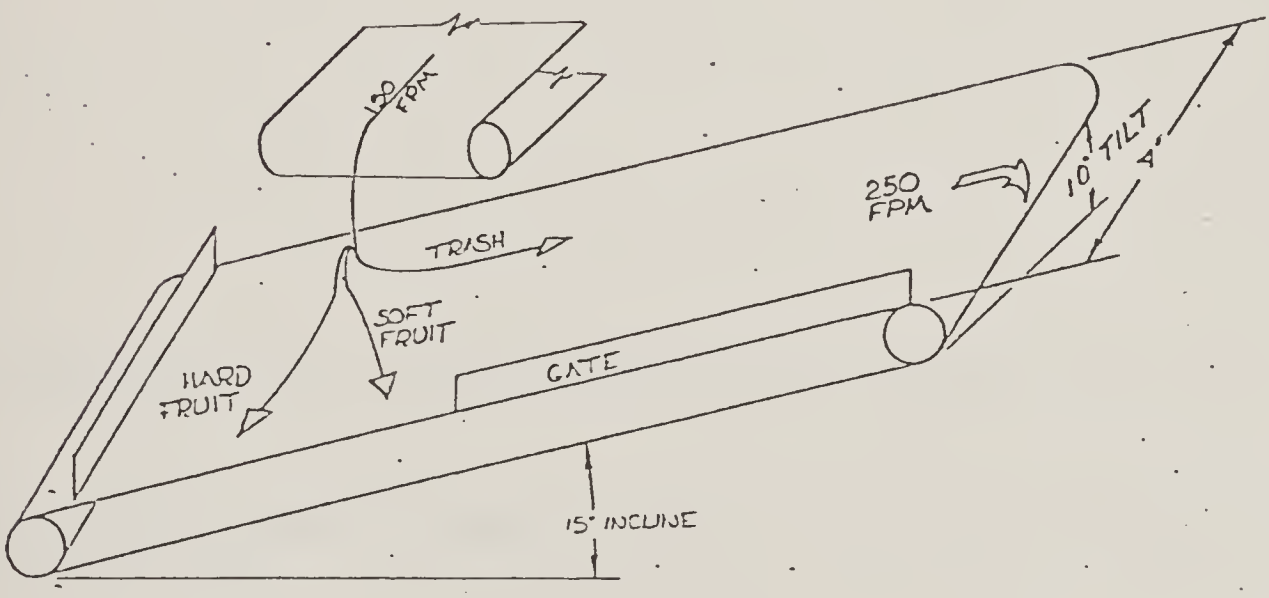

Figure 2. Pilot trash removal belt.

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